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Dichloro(6,6′-dimethyl-2,2′-bipyridyl)cobalt(II) Hemibenzene Solvate

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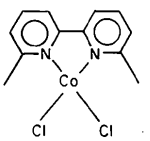
Abstract. $[CoCl_2(C_{12}H_{12}N_2)].\frac{1}{2}C_6H_6$, $M_r = 353{\cdot}14$, $P2_1/c$, monoclinic, $a = 7{\cdot}505$ (3), $b = 13{\cdot}544$ (3), $c = 15{\cdot}728$ (2) Å, $\beta = 96{\cdot}11$ (2)°, $V = 1589{\cdot}6$ (8) Å^3, $Z = 4$, $D_x = 1{\cdot}475$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 14{\cdot}1$ cm^{-1}, $F(000) = 720$, $T = 295$ (1) K, $R = 0{\cdot}028$ for 2103 reflections with $I > 3{\cdot}0\sigma(I)$ (2786 unique observations). The Co is tetrahedrally coordinated, with two essentially equal Co—Cl bonds [2·2139 (5) Å] and two equal Co—N bonds [2·040 (1) Å]. The five-membered metallocycle is in a slightly twisted envelope conformation, with Co in the flap [dihedral angle 3·8 (5)°]. A benzene molecule of solvation resides on the crystallographic inversion center.

Experimental. The compound (I) was synthesized as reported (Newkome, Pantaleo, Puckett, Ziefle & Deutsch, 1981). An aqua-blue, prismatic crystal was mounted with epoxy on a glass fiber in random orientation. Details of data collection and structural refinement are given in Table 1.



(I)

The structure was solved using the Patterson heavy-atom method which revealed the positions of Co and both Cl atoms. The remaining atoms were located in successive difference Fourier syntheses. H atoms were located and their positions and isotropic thermal parameters were refined. The structure was refined in full-matrix least squares with Enraf–Nonius *SDP* (Frenz, 1978) where the function minimized was $\sum w(|F_o| - |F_c|)^2$ and the weight w is defined as $4F_o^2\sigma^2(F_o^2)$. The final cycle of refinement included 241 variable parameters and converged to $R = 0{\cdot}028$. Atomic scattering factors, including those for anomalous dispersion, were taken from *International Tables for X-ray Crystallography* (1974).

Final positional and equivalent isotropic thermal parameters are given in Table 2, and selected bond

Table 1. *Experimental details*

Crystal	Blue, prismatic 0·16 × 0·32 × 0·52 mm
Instrument	Enraf–Nonius CAD-4 diffractometer
Monochromator	Incident-beam, graphite
Unit cell	25 reflections, 30·0 < 2θ < 31·7°
Mode	ω–2θ
Standards	200, 0$\bar{4}$0, 004
R_{int}	0·011
Corrections	Background, Lorentz, polarization
	Empirical absorption (0·867–1·000 on *I*)
	Linear decay (1·000–1·019 on *I*)
2θ range (°)	2·6–50·0
hkl ranges	$h = 0$ to 8
	$k = 0$ to 16
	$l = -18$ to 18
Reflections	3314 total
	2786 unique
	2103 with $I > 3{\cdot}0\sigma(I)$
Solution	Patterson method
Function minimized	$\sum w(\|F_o\| - \|F_c\|)^2$
Weights	$4F_o^2\mathrm{Lp}^2/[S^2(C+R^2B) + (0{\cdot}020F_o^2)^2]$;
	S = scan rate, C = integrated count,
	R = scan time/background time,
	B = background count
Parameters refined	241
R, wR, R(all)	0·028, 0·033, 0·050
Goodness of fit	1·88
Maximum shift/e.s.d.	0·05
$\Delta\rho$ (e Å^{-3})	0·30 (4), –0·16 (4)

Table 2. *Positional parameters and their e.s.d.'s*

The equivalent isotropic thermal parameter, for atoms refined anisotropically, is defined by the equation

$$\tfrac{4}{3}[a^2B_{11} + b^2B_{22} + c^2B_{33} + abB_{12}\cos\gamma + acB_{13}\cos\beta + bcB_{23}\cos\alpha].$$

	x	y	z	B_{eq}(Å^2)
Co	0·18404 (4)	0·08870 (2)	0·33539 (2)	4·008 (6)
Cl(1)	–0·06275 (9)	0·09453 (6)	0·24494 (5)	7·01 (2)
Cl(2)	0·40660 (9)	0·16774 (6)	0·28314 (4)	6·16 (2)
N(1)	0·1648 (2)	0·1237 (2)	0·4604 (1)	4·09 (4)
N(2)	0·2612 (2)	–0·0434 (1)	0·3894 (1)	3·76 (4)
C(1)	0·1147 (3)	0·2111 (2)	0·4909 (2)	5·27 (6)
C(2)	0·1212 (4)	0·2262 (2)	0·5790 (2)	6·49 (7)
C(3)	0·1776 (4)	0·1521 (2)	0·6340 (2)	6·56 (7)
C(4)	0·2291 (4)	0·0630 (2)	0·6028 (2)	5·33 (6)
C(5)	0·2208 (3)	0·0505 (2)	0·5151 (1)	3·97 (5)
C(6)	0·2717 (3)	–0·0433 (2)	0·4757 (1)	3·72 (4)
C(7)	0·3290 (3)	–0·1265 (2)	0·5222 (2)	5·00 (6)
C(8)	0·3749 (3)	–0·2094 (2)	0·4798 (2)	5·57 (6)
C(9)	0·3626 (3)	–0·2098 (2)	0·3923 (2)	5·45 (6)
C(10)	0·3050 (3)	–0·1256 (2)	0·3480 (2)	4·53 (5)
C(11)	0·0545 (5)	0·2877 (2)	0·4272 (2)	7·67 (9)
C(12)	0·2895 (5)	–0·1215 (2)	0·2530 (2)	7·27 (8)
C(1*B*)	0·4648 (5)	0·5244 (3)	0·4157 (2)	7·95 (9)
C(2*B*)	0·3274 (5)	0·5136 (3)	0·4638 (2)	8·15 (9)
C(3*B*)	0·6364 (5)	0·5117 (3)	0·4506 (2)	8·21 (9)

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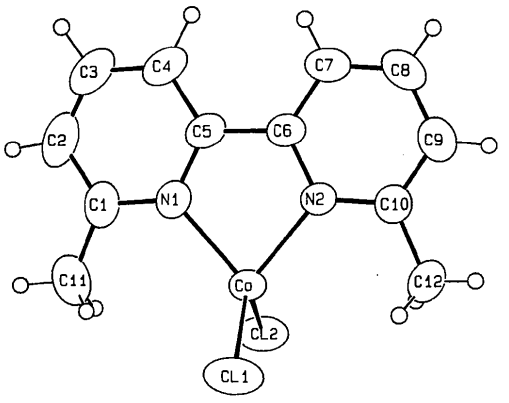

Fig. 1. View of the title complex with 50% thermal ellipsoids (Johnson, 1965).

lengths and bond angles are shown in Table 3.* Fig. 1 shows the molecule and the atomic numbering scheme.

Related literature. Preparation of Pd^{II}, Pt^{II}, Cu^{II} and Zn^{II} analogues: Newkome, Pantaleo, Puckett, Ziefle & Deutsch (1981); structure of Pd analogue: Newkome, Fronczek, Gupta, Puckett, Pantaleo & Kiefer (1982); structure of $[(C_{12}H_{12}N_2)_2Cu(I)]BF_4$: Burke, McMillin & Robinson (1980).

*Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond lengths, bond angles, torsion angles, and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51040 (34 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Selected bond lengths* (Å) *and angles* (°)

Co–Cl(1)	2·2134 (5)	N(1)–C(1)	1·347 (2)
Co–Cl(2)	2·2144 (5)	N(1)–C(5)	1·351 (2)
Co–N(1)	2·042 (1)	N(2)–C(6)	1·351 (2)
Co–N(2)	2·039 (1)	N(2)–C(10)	1·348 (2)
Cl(1)–Co–Cl(2)	110·89 (2)	Co–N(1)–C(1)	126·8 (1)
Cl(1)–Co–N(1)	118·07 (4)	Co–N(1)–C(5)	113·2 (1)
Cl(1)–Co–N(2)	118·72 (4)	C(1)–N(1)–C(5)	119·9 (2)
Cl(2)–Co–N(1)	112·24 (4)	Co–N(2)–C(6)	113·7 (1)
Cl(2)–Co–N(2)	112·78 (4)	Co–N(2)–C(10)	126·6 (1)
N(1)–Co–N(2)	81·28 (6)	C(6)–N(2)–C(10)	119·7 (2)

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Hygric Acid (I) and Stachydrine (II) as Their Hydrochlorides

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Abstract. (I) 1-Methyl-L-proline hydrochloride, $C_6H_{12}NO_2^+.Cl^-$, $M_r = 165·6$, orthorhombic, $P2_12_12_1$, $a = 6·717$ (3), $b = 10·397$ (1), $c = 12·140$ (1) Å, $V = 848$ (2) Å^3, $Z = 4$, $D_m = 1·28$, $D_x = 1·297$ Mg m^{-3}, Mo $K\bar{\alpha}$ radiation, $\lambda = 0·7107$ Å, $\mu = 0·346$ mm^{-1}, $F(000) = 352$, $T = 293$ (2) K, $R = 0·061$ for 750 observed reflections. (II) 2-Carboxy-1,1-dimethylpyrrolidinium chloride, $C_7H_{14}NO_2^+.Cl^-$, $M_r = 179·6$, orthorhombic, $P2_12_12_1$, $a = 6·561$ (2), $b = 11·671$ (6), $c = 11·690$ (4) Å, $V = 895$ (2) Å^3, $Z = 4$, $D_m = 1·31$, $D_x = 1·333$ Mg m^{-3}, Mo $K\bar{\alpha}$ radiation, $\lambda = 0·7107$ Å, $\mu = 0·331$ mm^{-1}, $F(000) = 384$, $T = 293$ (2) K, $R = 0·031$ for 1239 observed reflections. In the *N*-methylated and *N,N'*-dimethylated proline derivatives (I) and (II) the N atom lies out of the plane of the pyrrolidine ring, to the same side of the molecule as the

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